CHARACTERIZATION OF ACTIVATED CARBON OF COCONUT SHELL, RICE HUSK AND KARANJA OIL CAKE

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By

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CERTIFICATE

This is to certify that the thesis entitled "**Characterization of activated carbon of coconut shell, rice husk and karanja oil cake**", submitted by Smrutirekha Das, Roll No.-110CH0351, towards partial fulfillments of the requirement for the degree of Bachelor in Technology in Chemical Engineering at National Institute of Technology, Rourkela is prepared by her under my supervision and guidance.

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ABSTRACT:

The studies on characterization of different type of adsorbent such as coconut shell activated carbon, activated carbon from rice husk and activated carbon from karanja oil cake has been performed. The characterization of the adsorbents includes estimation of various parameters such as proximate analysis (moisture content, ash content, volatile matter content and fixed carbon content), bulk density, BET surface area, SEM, porosity, pH, iodine number and methylene blue number. Surface area of adsorbents was found by BET surface area analyzer. The pore structure of activated carbon was observed through SEM analysis. The porosity and pore volume was estimated using mercury porosimeter. The adsorbent that showed best surface properties was used for adsorption of methylene blue dye. Adsorption capacity of the activated carbon was determined to access its maximum potential for methylene blue removal.

table of contents

LIST OF TABLES

LIST OF FIGURE

NOMENCLATURE

^0C	: Degree Celsius
BET	: Brunnauer Emett Teller
nm	: Nanometer
SEM	: Scanning electron microscope
ASTM	: American Society for Testing and Materials
M	: Moisture content
A	: Ash content
VM	: Volatile matter
FC	: Fixed carbon
P_0	: Saturation pressure of the adsorbate
P	: Equilibrium pressure of the adsorbate
v	: Volume of gas adsorbed
v_m	: Volume of gas adsorbed in the monolayer
c	: BET constant
E_1	: Heat of adsorption for the first layer
E_L	: Heat of adsorption for higher layers
R	: Universal gas constant
T	: Absolute temperature
N	: Avogadro's number
s	: Adsorption cross-section of the gas being adsorbed
V	: Molar volume of the gas being adsorbed
X	: Mass of the adsorbent
C_0	: Initial concentration
C_e	: Equilibrium concentration
q_e	: Adsorption capacity

CHAPTER – 1

INTRODUCTION

Introduction:

Increase in population has boosted the growth of different industries leading to discharge of pollutants into the water bodies. Among those industries textile, food, cosmetic, paper industries lead to discharge of dye that needs immediate attention. Colour in the water results from various organic chemicals that prevent the sunlight to penetrate affecting the aquatic system. Aquatic organisms and plants are affected due to the release of toxic organic chemicals.

Various methods to address this issue has been published by many researchers such as sedimentation with clarification, coagulation and flocculation, chemical oxidation, filtration using membranes, adsorption, biodegradation etc . Among these adsorption is a well established technology to deal with dye removal. Methylene blue dye has been used in most of the industries and its removal is a matter of great concern. Low cost adsorbents such as coir pith, sawdust, fruit shell, banana pith, pea nut hull, wheat barn etc has been employed [Vadiyelan et al, 2005; Chandran et al, 2002; Bhattacharya et al, 2005; Kumar et al, 2005; Garg et al, 2003; Hamdaoui et al, 2007]. However due to its less adsorption capacity use of activated carbon as an adsorbent is greatly sorted.

Activated carbon is a special type of carbonaceous substance. It has highly crystalline form and extensively developed internal pore structure. Due to activation, internal pore network is created which imparts certain surface chemistries (functional groups) inside each particle. Thus carbon gets its unique characteristics leading to high surface area, porosity and greater strength.

The adsorptivity of the adsorbent depends on both the size of the molecule being adsorbed and the pore size of the adsorbent.

The organic material which has high carbon content is used as the raw material for the synthesis of activated carbon. There are many cheap, easily available such as wheat husk, straw, palm fiber, rubber wood saw dust, bamboo dust, date pits, palm fiber, coconut shell, groundnut shell, oil cake etc have been used as the source for the synthesis of activated carbon.

They are used in the abatement of hazardous contaminants, treatment of municipal and industrial waste water, as catalyst or catalyst support in medicine, and the recovery of valuable metals

Thus the aim of this research is to look for a good adsorbent based on their surface characteristics. It also explores the adsorption capacity of the adsorbent for methylene blue dye removal.

Objectives:

(1) Characterization of activated carbon which is synthesized from coconut shell, rice husk and karanja oil cake.

(2) Compare the characteristics of coconut shell activated carbon with other two.

(3) Predict which activated carbon is better over other two for removal of methylene blue dye.

CHAPTER-2

LITERATURE REVIEW

Literature Review:

Many experiments and studies have been carried out regarding the characterization of the activated carbon which was synthesized from source such as fluted pumpkin seed, groundnut shell, palm oil waste, karanja oil seed, rubber wood saw dust etc.

Table -1 list of different type of activated carbon which was taken for characterization

Different type of activated carbon using different precursors	Authors
Fluted pumpkin seed shell	Verla, A.W, M.Horsfall, E.N Verla, A.I. Spiff, O. A. Ekpete, 2012
Biomass waste	A S Jadhav, Sandeep Salwa, MangeshTambe, N H shinde, 2011
Groundnut shell	R Mallik, D S Ramteke& SR Wate, 2006
Activated carbon	Hary Marsh, FranciseoRodriguer- Reinoso, 2006
Rice husk & palm fruit shell	JEG Mdoe and L Mkayula, 2002
Oil palm kernel shell & oil palm fibre	Evbuomwan B.O, Agbede A.M, Ataka M.M,2013
Olive cake	Yak out S.M., Sharf, 2011
Rice husk ash	Otaru,A.J., Amech, C.U.,A.S, A.S, OkaforJ.O,Abdulkareem, A.S, and Odigure, J.O 2013

P.K Mallick , 2004 used Mahogany sawdust to develop an effective carbon adsorbent. This adsorbent was employed for the removal of dyes from spent textile dyeing wastewater. The experimental data were fitted to Langmuir and Freundlich models of adsorption.

M.M.Nourouzi and T.G.Chuah in 2009 studied the adsorption behavior on Reactive Black 5 and Reactive red 3 using Palm Kernel Shell Activated carbon. Applications of batch kinetic data to pore and film surface diffusion models were explored.

Jun –jie Gao et al., 2013 produced activated carbon from tea seed shells. They obtained activated carbon of BET surface area 1530 m^2/g. The precursor was chemically activated using zinc chloride and pyrolysed in a tubular furnace at 500^oC for one hour duration at a heating rate $5^oC/min$.

Halandemiral et al., 2008, prepared activated carbon from Hazelnut bagasse through chemical activation technique. The surface area developed was significant 1489 m^2/g. It was employed to remove Sandolan blue from the water bodies.

There were some experiments on characterization of different type of activated carbon which were given in the previous table. These activated carbon materials are characterized by their large surface areas and better porosity which was well developed. For these reasons activated carbons are commercially used as adsorbents for the removal of some organic chemicals and metal ions of environmental, potable water, waste water and removal of some gases.

Usually the cost of preparation of activated carbon depends on the selection of the precursors.. So from the economical view, to utilize the cheaper raw materials for the production of activated carbon is sorted. Activated carbons are widely used as an economic and stable mass separation agent final or the removal of dye or surfactants and improve the quality of the final product in many industrial processes. Activated carbon is also used for the purification of liquids and gases, separation of mixtures and catalysis. Adsorption capacity of activated carbon is measured by considering the chemical nature of the aqueous phase, the solid phase and the chemical nature of the adsorbing organic.

For the analysis of the surface physical properties of the carbon which includes the determination of the total surface area, extent of micro-porosity and characterization of the pore distribution. The total surface area can be measured by using BET method and pore structure can be shown by SEM test. Pore volume and porosity can be obtained from porosimetry. The adsorbent having large pore volume is used as better adsorbent for the adsorption process.

CHAPTER-3

MATERIALS AND METHODS

3.1.1 Raw material:

For this project the raw materials such as coconut shell activated carbon, activated carbon from rice husk and activated carbon from karanja oil cake was obtained.

3.1.2 Chemicals:

The various chemicals used during this project were hydrochloric acid, sodium thiosulphate, iodine solution, starch, 1N sulfuric acid, 1N sodium hydroxide etc.

3.2 Instrumentation:

Muffle furnace, oven, weighing machine, Brunaner Emmett Teller surface area analyzer, pH meter, mercury porosimetry, scanning electronic microscope, hand shaker, UV spectro-photometer.

3.3 Proximate analysis:

According to ASTM D 121, proximate analysis is the determination of moisture, volatile matter, fixed carbon, and ash content by prescribed methods. The proximate analysis of the different type of activated carbon was done using the following procedure.

3.3.1 Moisture content:

Small amount of activated carbon sample (coconut shell) weight was measured and then taken in a petri dish. It was spread nicely on the dish. It was then heated in an oven at a temperature of (105-110)˚c for 1.5hr. The petri dish was left open or not covered during heating process. After heating petri dish was removed and cooled in a desicator. After cooling the weight of dried sample was measured.

Fig-1 laboratory hot air oven

Moisture content M=100(B-F)/(B-G)

B=weight of petri dish +original sample

F=weight of petri dish+ dried sample

G= weight of petri dish

Same procedure was also followed for determination of moisture content for activated carbon of rice husk and karanja oil cake.

3.3.2 Ash content:

1 gm of sample was taken in a silica crucible. It was heated in a muffle furnace to 750˚c for 1.5hr. During this heating process the crucible was left open. After the required heating, the crucible was cooled in a desicator and then weight of the ash was measured.

Ash content A=100(F-G)/(B-G)

G=Mass of empty crucible

B=Mass of crucible + sample

F=Mass of crucible+ ash sample

Same procedure was also followed for determination of moisture content for activated carbon of rice husk and karanja oil cake.

3.3.3 Volatile matter content:

A known quantity of sample was taken in cylindrical crucible closed with a lid. It was then heated to 925˚c for exactly 7minutes in a muffle furnace. Then the crucible was cooled in a desicator and weighted.

Volatile matter on dry basis

VM=100[100(B-F)-M(B-G)]/[(B-G)(100-M)]

Fig-2 muffle furnace

B=Mass of crucible, lid and sample before heating

F=Mass of crucible, lid and contents after heating

G=Mass of empty crucible & lid

M=% of moistures determined above

Same procedure was also followed for determination of moisture content for activated carbon of rice husk and karanja oil cake

3.3.4 Fixed carbon content:

Fixed carbon FC = 100 – (% moisture content+ % volatile matter + % ash content)

3.4 Bulk Density:

Bulk density is defined as weight per unit volume of material. It is basically used for powder of materials. This test of bulk density represents the flow consistency and packaging quantity of solid sample. It is expressed in terms of kilograms per cubic meter (kg/m^3). First measure the mass of the measuring cylinder which was used for this experiment. Then the given sample of activated carbon was placed into this cylinder and reweighed it. Then it was transferred into the aluminium plate and then oven dried at a temperature of 105˚C for 60mins. After drying the weight of the dry sample was measured.

D_B= (m2- m1)/v

M1= mass of measuring cylinder in grams

M2= mass of measuring cylinder + its contents

V= volume of the measuring cylinder in litre

3.5 BET surface area:

Brunauer, Emmett and Teller-are the three men who proposed a theory to measure the surface area of porous powder type solid particles. The principle involved is the adsorption of gas molecules to the surface of the solid whose surface area is required. From the area of each molecule, the whole area of the solid can be calculated.

BET theory is based on multilayer adsorption with considering the following assumptions:

- Gas molecules can be physically adsorbed on the solid surface and form infinite layers
- There is no interaction between layers
- Langmuir theory is applied to each layer

The BET equation is given by:

$1/v[(P_0/P)-1]=(c-1)(P/P_0)/(v_m c)+1/(v_m c)$

Where,

P_0=Saturation pressure of the adsorbate

P = Equilibrium pressure of adsorbate

v = Volume of gas adsorbed

Fig-3 BET surface area analyzer

v_m= volume of the gas adsorbed by the monolayer

c = BET constant given by exp (E_I-E_L)

E_I=Heat of adsorption for the first layer

E_L=Heat of adsorption for higher layers

A plot of $1/v[(P_0/P)-1]v/s(P/P_0)$ is obtained from the BET analysis. From the slope and intercept of the line, v_m and c are obtained. Surface area S of the solid sample is given by

$S=(v_mNs)/(vx)$

N=Avogadro's number

S= adsorption cross section of the gas being adsorbed

v = molar volume of the gas being adsorbed

x = mass of the adsorbent

A small amount of the adsorbent sample was taken in the tube and the tube was placed in a dewar containing liquid nitrogen. Initially the sample was degasified to remove the impurities and gases. Then gaseous nitrogen was passed through the sample and based on adsorption of the gas, the surface area of the sample was calculated. This procedure is same for all three different type of activated carbon.

3.6 Mercury porosimetry

Mercury porosimetry is an effective instrument for the measurement of porosity and pore volume of porous substances. Initially gas, moisture and other impurities were evacuated from the sample and vacuum is created. Mercury was transferred into the sample under vacuum and gradually the pressure was increased. Amount of mercury intruded was measured continuously which was a function of applied pressure. Further analysis of this gave an intrusion extrusion curve and parameters describing the pore structure were calculated from the data.

The activated carbon of sample was placed in the vacuum chamber and the procedure as mentioned above was applied to measure the desired parameters. Repeat the procedure for other two type of activated carbon.

3.7.pH

pH was determined using the standard method ASTMD 3838-80. 1g activated carbon of coconut shell sample was put in a conical flask and 100ml distilled water was added to it. The mixture was stirred for1hour. pH readings were taken using pH meter. Repeat the procedure for the other two type of activated carbon.

Fig-4 pH meter

3.8 Iodine number:

Iodine number is the milligrams of iodine adsorbed by 1gram of activated carbon from a standard 0.1N iodine solution when the equilibrium iodine concentration is exactly 0.02N. Iodine number is a measure of the micro-pore content of the activated carbon. A higher iodine number signifies higher micro-porosity of the sample.

ASTMD4607 –94(2006) gives the standard procedure for the determination of the iodine number of the activated carbon.0.7-2gof dried activated carbon was mixed with10ml of 5% by weight of hydrochloric acid in a conical flask. The nit was swirled until the activated carbon was wetted. The conical flask was boiled for 30sec not directly but by placing it on a hot plate. The contents of the flask were cooled to room temperature and then 100ml 0.1N iodine solution was added to it. The flask was shaken vigorouslyfor30 sec. The contents were filtered through a filter paper. Initially 20-30ml of the filtrate was discarded and the remaining filtrate was collected in a clean beaker. Then 50ml of this filtrate was titrated against0.1Nsodium thiosulphate solution until yellow color just disappeared. After that 1ml starch solution was added into it and titration was continued till blue color just disappeared. Concentration of the final solution was calculated and if the equilibrium concentration is not within 0.008 to 0.0334, the procedure is repeated with a different amount of activated carbon. This procedure is same for the three different type of activated carbon.

3.9 Methylene blue number:

Methylene blue number of activated carbon is correlated with ability of the activated carbon to adsorb colour and high molecular weight substances. To determine the methylene blue number of the activated carbon, 50 ml of 200 ppm methylene blue solution was shaken for 30 minutes in a 500 ml Erlenmeyer flask containing 0.1 g activated carbon and then was filtered using a membrane filter. The color of the filtrate was measured in a UV-Vis spectrophotometer and compared with a control.

3.10 Comparison study of adsorption capacity of different type of activated carbon:

A 100ml solution of methylene blue with a concentration of 50ppm was taken in a conical flask. Then check the pH of the solution. Make it up to pH of 5 by adding either 1N sulfuric acid or by adding 1N sodium hydroxide by our requirement. Then 0.6gm of activated carbon (coconut shell) was added into it and then kept the solution into the hand shaker for the period of 60 minute.

Fig- 5 Spectrophotometer

After that take the solution and measure the absorbance after adsorption by using UV-spectrophotometer and determine the final concentration of the solution. Then calculate the percentage removal of methylene blue. Repeat the procedure for other two adsorbent. Now compare the adsorption capacity of given three type of activated carbon. And choose the best one adsorbent over other two.

3.11 Scanning Electronic Microscope (SEM):

Scanning electron microscope is a type of microscope which is used for visualization of porous structure of a material. It uses a beam of highly energetic electrons to scan a sample and create its image. Electron gun acts as a source for electrons here. The electron beam is focused by a pair of condenser lenses made of magnets. These magnets are capable of bending the path of electrons. Sample was placed in the sample chamber for analysis. The electron beam strike the sample, get decelerated. It produced a variety of signals like secondary electrons, back scattered electrons, diffracted back scattered electrons, photons, visible light and heat. The secondary electrons were picked up by the detectors and produced the images of the object’s surface on the monitor. The entire operation took place inside a vacuum chamber. The activated carbon sample was analyzed in a SEM to visualize the porous structure. The magnification was adjusted for getting a clear picture

Fig-6 SEM set up

CHAPTER-4

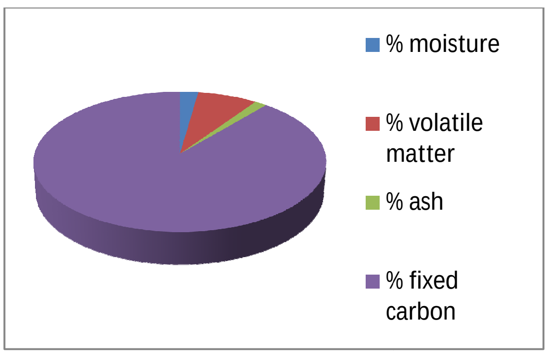
RESULTS AND DISCUSSION

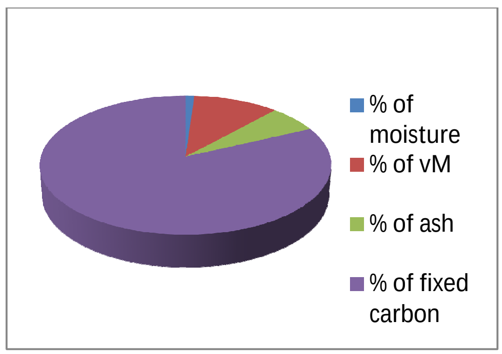
Results and discussion:

4.1 Proximate analysis:

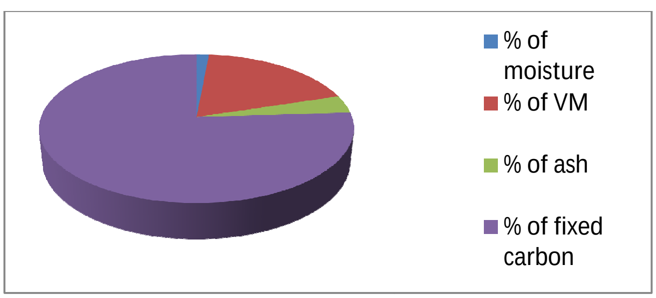
Table – 2 Proximate analysis of activated carbons

	Coconut shell AC	Rice husk AC	Oil cake AC
% moisture content	2.22	1.07	1.33
% VM content	7.358	14.62	18.67
% ash content	1.5	6.25	3.92
% FC content	88.922	78.06	76.08





(a) (b)



(c)

Fig-7 (a), (b) & (c) Proximate analysis of activated carbons from coconut shell, rice husk, oil cake respectively

It helps us to assess the amount of moisture, volatile and ash content with the residual carbon present in the sample It shows that the moisture content of activated carbon prepared from coconut shell is high while the volatile and ash content are the least. Due to its high fixed carbon it is preferred adsorbent with respect to other two

4.2 Bulk density:

It provides a bird eyeview regarding the floatability property of the adsorbent. It suggests that if the activated carbon is added to water it will sink and that with result in better contact with the adsorate and thereby leading to effective adsorption process.

Table -3 Bulk density of activated carbons

Type of AC	Bulk density (gm/cc)
Coconut shell	0.58
Rice husk	0.121
Oil cake	0.352

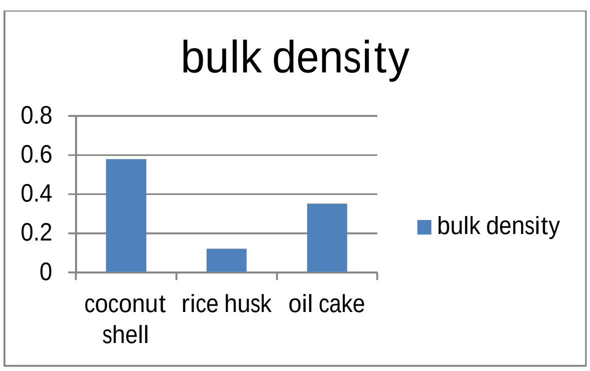


Fig – 8 Bulk density of different type of activated carbon

4.3 Iodine Number:

Table – 4 Iodine number of different type of activated carbon

Type of AC	Iodine number
Coconut shell	986
Rice husk	178
Oil cake	258

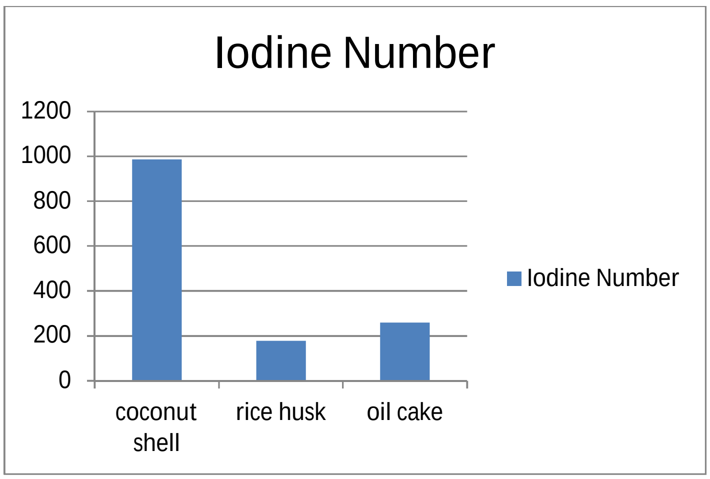


Fig – 9 Iodine number of activated carbon

From this graph we can observed that the iodine number of activated carbon of coconut shell was highest value. It signifies that coconut shell has more micro pores. Due to high pores it acts as better adsorbent for adsorption process.

4.4 Characterization of activated carbon:

All the characterization of activated carbon of coconut shell, rice husk and karanja oil cake shown in the following table.

Table- 4 Compilation of characteristics of three type activated carbon

characteristics	**Coconut shell AC**	**Rice husk AC**	**Oil cake AC**
% moisture content	2.22	1.07	1.46
% volatile matter	7.358	14.62	18.67
% ash content	1.5	6.25	3.92
% fixed carbon	88.922	78.06	76.08
Bulk density (gm/cc)	0.58	0.121	0.352
BET surface area (m^2/g)	1000.15	289.1	179.3
pH	6.52	5.7	6.2
Iodine number(mg/g)	986	178	258
Methylene blue number(mg/g)	226	52.7	105.3
Pore volume (cc/gm)	0.07	0.0537	0.0592

Ash is non-carbon or mineral additives, that does not combine chemically with the carbon surface. It consists of various undesired mineral substances, which become more concentrate on activation and comprises of 1-20 % and primarily depends on the type of raw material.

High ash content is undesirable for activated carbon since it reduces the mechanical strength of carbon and affects adsorptive capacity. Iodine adsorption is a simple and quick technique to determine the adsorptive capacity of activated carbon, also known as iodine number. It was observed that iodine number was maximum for coconut shell activated carbon that indicates maximum surface area according to [Puri and Bansal; 2001].

Similarly methylene blue value helps us to determine the surface area and the pore size distribution of activated carbon. The methylene blue value represent the adsorptive capacity of activated carbon for molecules with dimension similar to methylene blue and the surface area which results from the presence of pore sizes greater than 1.5 nm. The methylene blue value for coconut shell activated carbon was maximum with respect to rice husk and oil cake activated carbons.

4.4 **Adsorption capacity of each activated carbon:**

Table-5 adsorption capacity of 3 type of activated carbon

Type of AC	Initial dye concentration(mg/Lt)	Final dye concentration(mg/Lt)	% of removal of blue methylene	q□ = (C□-C□)/x (mg/g-Lt)
Coconut shell	50	2	96	80
Rice husk	50	19	62	51.67
Oil cake	50	8.5	83	69.17

From the above table we can observe that the adsorption capacity of activated carbon of coconut shell is better than the other two.

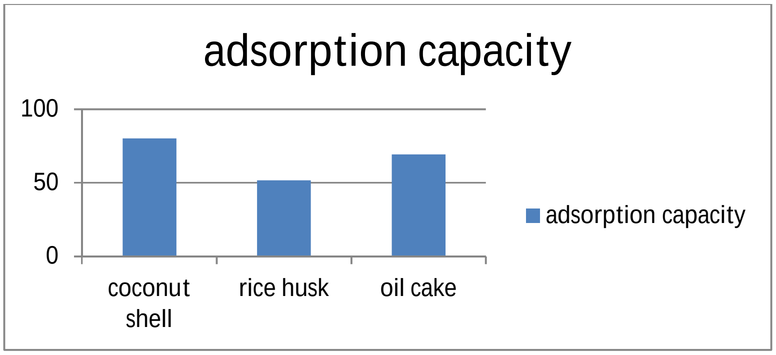


Fig -10 adsorption capacity of different type of activated carbon

4.5 **SEM of coconut shell activated carbon:**

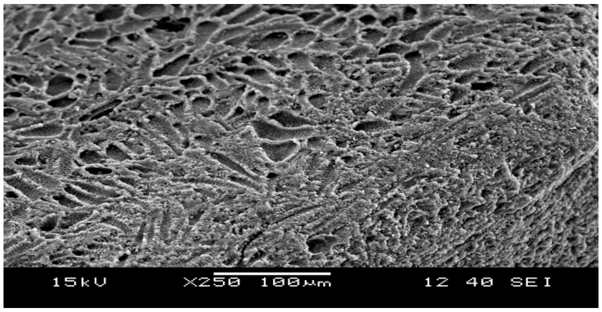


Fig-10(a) SEM image of activated carbon at X250 zoom

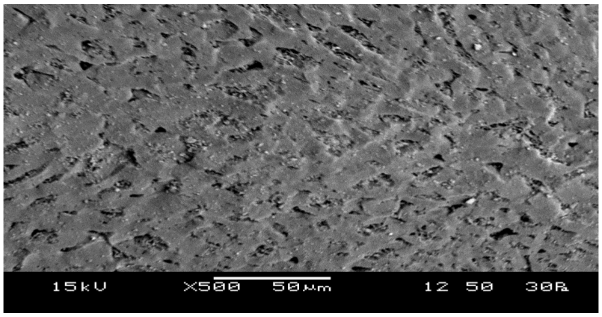


Fig- 10(b) SEM image of activated carbon at X500 zoom

Scanning electron microscope images were taken to observe the surface topography of the sample. Basically, the pore structures of the activated carbons were observed. The micrographs of the activated carbon are shown in above.

CHAPTER -5

CONCLUSIONS

Conclusions:

The study required to conduct many characterization techniques like proximate analysis, bulk density, BET surface area, pH, Pore volume, iodine number test, blue methylene number test. This is followed by an adsorption experiment to find the better adsorbent for adsorption process. Then SEM test of better adsorbent was performed on the adsorbent and show the pore structure of that adsorbent.

Proximate analysis of activated carbon provides a good idea about the physical properties of sample. From this analysis it is found out that, the fixed carbon content of coconut shell activated carbon is higher than other two. It shows that the moisture content of activated carbon prepared from coconut shell is high while the volatile and ash content are the least. This is due to its high fixed carbon, which is a preferred adsorbent in comparison to other two. From the bulk density test we calculated and observed that the bulk density of coconut shell is higher compared to the other two thus implying better flow consistency and packaging quantity of solid fuel material.

From the experiments it is found that the BET surface area of coconut shell 1000.15m^2/gm which is more than the rice husk and the oil cake. From iodine number test we found that the iodine number of coconut shell is 986 mg/gm. This result indicates which sample is better over other two. Iodine number is basically a measure of the micro-pore content of the activated carbon. Iodine number shows a direct proportionality with the micro porosity of the sample, thus, a higher iodine number signifies higher micro-porosity of the sample. Pore volume of the coconut shell activated carbon is larger than the other two.

From methylene blue number test we found that the methylene blue number of coconut shell 226 mg/g which is higher than the other two. From the adsorption test, we found that coconut shell activated carbon gives the better adsorption of methylene blue dye over the rice husk and oil cake.

Following the characterization tests where we found preferable and desired results for the coconut shell activated carbon, the SEM test was carried out to observe pore structure of better adsorbent activated carbon.

CHAPTER-6

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